

**MISSOURI DEPARTMENT OF NATURAL RESOURCES  
AIR AND LAND PROTECTION DIVISION  
ENVIRONMENTAL SERVICES PROGRAM  
Standard Operating Procedures**

SOP #: MDNR-FSS-011 EFFECTIVE DATE: January 3, 2003

SOP TITLE: General Sampling Considerations for Chemical Analysis of Sediments

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SUMMARY OF REVISIONS: Not applicable, this is a new SOP

APPLICABILITY: The methods discussed herein are applicable to all ESP personnel who are involved in any type of sediment sampling where verifiable and defensible field and analytical data are required.

DISTRIBUTION: Supervisor, FSS, WQMS  
MoDNR Intranet  
ESP SOP Coordinator

RECERTIFICATION RECORD:

Date Reviewed				
Initials				

## 1.0 SCOPE AND APPLICABILITY

- 1.1 Assessment of water quality is a major function of the Environmental Services Program (ESP). "Sediment contamination is one of the major end results of pollutant discharges into freshwater and marine environments. For many toxic materials, sediments represent both the primary repository and in many cases the principle source of contamination to the food chain." (Landrum and Robbins, 1990). These toxic materials include numerous organic compounds and heavy metals that may bioaccumulate within benthic organisms and pose a threat to fish and wildlife and a health hazard to humans.
- 1.2 Accurate and precise measurement of sediment associated contaminants requires standardized procedures for conducting sediment sampling investigations. This ESP document provides guidelines for proper sampling of sediments.

## 2.0 SEDIMENT SAMPLING STUDIES

- 2.1 The ESP conducts sediment studies to determine the presence, concentration, and distribution of sediment contaminants within lakes and streams. When feasible, sediment studies are conducted in conjunction with toxicity testing, aquatic macroinvertebrate studies, fish tissue studies, and physiochemical water quality analysis.
- 2.2 Most ESP sediment studies focus on the effects of hazardous substance spills, such as crude oil, upon sediments, biota, and water quality. Other investigations may be concerned with longer-term releases of toxins that may be bound to or accumulate in sediments such as leachates from landfills, mines, hazardous waste sites, and product or process releases from point source discharges such as wastewater or industrial treatment facilities. Sediment may also be collected and analyzed for toxicity and a wide variety of contaminants as part of a long-term ambient monitoring project.

## 3.0 SAMPLING CONSIDERATIONS

- 3.1 Before any sediment study is undertaken, a sampling plan should be written. A reconnaissance survey is often necessary to define specific sampling strategies. Items that should be incorporated into a sampling plan include: review of site and previous work; scope and objectives; personnel and equipment; statistical strategy and sampling scheme; quality assurance/quality control procedures; safety considerations; and decontamination procedures. For detailed consideration of sampling, refer to the following: ESP Standard Operating Procedures and Project Procedures; Ecological Assessment of Hazardous Waste Sites: A Field and Laboratory Reference (USEPA, 1990); Standard Methods for the Examination of Water and Wastewater (APHA, 1995); and, Sediments: Chemistry and Toxicity of In-Phase Pollutants (Geisy and Muntau, 1990).

- 3.2 If dry weight analysis is needed, request “dry weight analysis” by writing it in the Analyses Section of the chain-of custody form (MDNR-FSS-002). Other analyses such as toxicity and ammonia content may be conducted on interstitial (pore) water.

#### 4.0 SAMPLE SITE SELECTION

- 4.1 See the ESP Standard Operating Procedure MDNR-FSS-005 for general sampling considerations. The selection of sediment sampling sites will be dependent on study objectives. Most importantly, sample locations must be representative sites. For example, a sediment study of a contaminant that is restricted to a particular stratum or sediment type must be limited to those variables in order to produce valid results. In addition to characteristics of the pollutant, several other factors will determine the selection of sampling locations. Some of these are: the type of receiving water, mode of entry of the contaminant, watershed and land use, population centers and water withdrawals, point and nonpoint discharges, and tributaries.
- 4.2 Another important factor in site selection is the availability of fine sediment. Sites more likely to have fine sediments should be found and selected for sampling. Sites unlikely to have adequate deposits of fine sediments include high gradient stream sections, stream headwater sections, channelized stream segments, and Ozark-type (gravel-bottomed) streams.

#### 5.0 STATISTICAL STRATEGY/SAMPLING SCHEME

- 5.1 Simple (or restricted) random sampling may be used when there is little knowledge of the distribution of the sample parameter or when subdivisions of the sample area are not needed. The sampling design is set up so that every unit of the population (e.g. a point on a grid) has an equal chance of being selected. Typically, however, contaminants or parameters within sediments are not evenly distributed and therefore may not provide meaningful data.
- 5.2 Stratified random sampling may be used in quantitative studies when the distribution of the parameter can be subdivided within the population or sampling site. For example, most sediment contaminants are stratified or more concentrated within fine sediments that have high organic content. Within the selected strata at each location, simple random sampling techniques can be employed. Reconnaissance or pilot studies should be conducted prior to planning a stratified sampling program.
- 5.3 Qualitative sediment sampling is commonly employed when statistical inference is not necessary. Qualitative sampling may be either simply judgmental such as documentation of sediment contamination or it may be systematic. Qualitative systematic sampling is often used when it is desired to intensively sample all

possible strata at a location. Qualitative comparative studies and many reconnaissance studies are examples of this type of sampling scheme.

## 6.0 HEALTH AND SAFETY REQUIREMENTS

- 6.1 Collector's supervisor should be notified of sampling plans including dates and locations prior to sampling trip.
- 6.2 Proper protective gear including clean elbow length rubber gloves should be worn during sampling and transfer to containers. Protective eyewear should be worn at least during equipment decontamination to protect eyes from acids and/or solvents.
- 6.3 When travelling and sampling by boat, all required safety gear should be on board including, personal floatation devices, sounding device, flares, fire extinguisher, cell phone, anchor, and oars. Personal floatation devices should be worn at all times while on board watercraft.
- 6.4 While operating a grab sampler, use of a protective back brace is recommended.
- 6.5 Field personnel shall have at a minimum attended department-sponsored inspection and enforcement training or received training from an ESP employee knowledgeable of sediment sampling procedures. Other training personnel should receive includes operation of watercraft, if necessary, and HAZWOPER and eight hour refresher courses if sampling for possible hazardous materials.

## 7.0 SUPPLIES AND EQUIPMENT

- 7.1 Three general types of sediment sampling equipment (scooping devices, core samplers, and grab samplers) may be used by sampling personnel depending on water depth, sediment characteristics, and study objectives.
  - 7.1.1 Scooping devices may be any small utensils shaped like a ladle, trowel, or spoon. Non-corrosive stainless steel spoons are to be used by the ESP as scooping devices. They should only be used in still water that is six inches or less in depth. Even in still water, some loss of surficial sediment may occur during transfer to sample container. Also, it is difficult to collect sediments at prescribed depths using a spoon or trowel. For these reasons, scooping devices are limited to shallow sediments.
  - 7.1.2 Core samplers (Appendix A) consist of a non-corrosive stainless steel cylindrical pipe (usually) or pipes that is/are pushed into the sediment (hand corer) or allowed to drop through the water column into the sediment (gravity corers). Extension rods on hand corer samplers can be used to collect sediments from water depth of up to fifteen feet. Gravity

corers can be used at greater depths, but are not as controllable in conditions such as high flows or choppy surface. Check valves at the bottom and top of the pipe prevent loss and disturbance of sediment upon retrieval. Core samplers are used to profile stratification of sediments and may be used for qualitative or quantitative sampling.

7.1.3 The grab samplers ESP uses are the Ponar (full size) and Petite Ponar (Appendix B). Grab samplers are designed to collect a consistent area of bottom sediments. They are usually lowered via a rope or cable to the bottom where gravity or a spring release actuates closure of the sampler. The only depth limitations of the Ponar samplers are length of rope or cable and the ability to control the sampler in a vertical position. The Ponar samples an area of 81 square inches and the Petite Ponar samples an area of 36 square inches. Both samplers can be used for a wide variety of sediments including mixtures of silt, sand, and small gravel. Because of their wider application and construction of stainless steel, they are generally preferred over other grab samplers. The full size Ponar is recommended for use in deeper water with higher flows such as the Missouri and Mississippi Rivers. Grab samplers must be lowered slowly into sediments to minimize disturbance of surficial layers. Grab samplers should not be used to collect sediment profiles. They may be used for qualitative or quantitative sampling.

## 7.2 Other sampling equipment

- solid braided nylon rope (for use with Ponar and Petite Ponar)
- decontaminated stainless steel collection bucket and spoon or other scooping device
- wading rod or other sediment detection device

## 7.3 Supplies

- pre-cleaned glass jars
- sample labels
- chain-of-custody record
- cooler/ice
- indelible ink pen
- maps
- field notebook
- elbow length rubber gloves
- paper towels
- GPS unit
- waders/boots

#### 7.4 Decontamination kit

- scrub brush
- phosphate-free detergent
- HCL or HNO<sub>3</sub> (5 or 10%)
- acetone
- deionized water

### 8.0 SAMPLING PROCEDURES

#### 8.1 Scooping devices

- 8.1.1 When collecting sediments for volatile organic analysis (VOA), refer to MDNR-FSS-006B.
- 8.1.2 Insert pre-cleaned, decontaminated scoop into sediment and slowly remove sample so as to disturb sample as little as possible.
- 8.1.3 Use a decontaminated stainless steel bucket for mixing sediments. Unless sampling for volatiles, homogenize until there is a consistent color and composition of the sediment. If sampling for volatiles, do not mix.
- 8.1.4 When filling multiple jars, to ensure each container receives a representative portion, fill each jar half full, then go back and fill jars completely. Clean outside of jars and secure lids of jars tightly.
- 8.1.5 Preserve, handle, and document as per Section 9.0.
- 8.1.6 Decontaminate equipment as per Section 10.0.

#### 8.2 Core sampler using hand coring method.

- 8.2.1 When collecting sediments for VOA, refer to MDNR-FSS-006B.
- 8.2.2 Insert a pre-cleaned, decontaminated core liner (Appendix A) into the sampler.
- 8.2.3 Add an extension rod to the corer to allow hand coring in water depths up to fifteen feet if necessary.
- 8.2.4 Force a pre-cleaned, decontaminated core sampler into the sediment to the desired depth, twist one-quarter turn and withdraw corer with a smooth continuous motion.

- 8.2.5 Remove the nosepiece (Appendix A) at the end of the core sampler and withdraw sample into a decontaminated stainless steel bucket. Use the core removal tool, if necessary, to aid in removal of the sample from the liner.
- 8.2.6 If a composite sample is required, rinse sediment from the corer after each grab with surface water at the sample site. The same core liner and core catcher may be used to collect a composite sample.
- 8.2.7 If particular strata of sample are required, measure down from the top of the core sampler to the desired depth and remove strata from corer with stainless steel or Teflon spoon.
- 8.2.8 Mix and transfer as per Sections 8.1.3 and 8.1.4 with decontaminated spoon.
- 8.2.9 Preserve, handle, and document as per Section 9.0.
- 8.2.10 Decontaminate equipment as per Section 10.0.

### 8.3 Core sampler using gravity coring method

Gravity coring is identical to hand coring except that a core sampler is allowed to drop via a rope to the bottom of the water body. Weights may be attached to the core sampler to sink it and push it into the sediment. Lower the corer slowly to minimize disturbance of the sediment. Gravity coring is used when the water depth exceeds the capacity for hand coring which is typically at fifteen feet or deeper.

### 8.4 Ponar or Petite Ponar grab sampler

- 8.4.1 When collecting sediments for VOA, refer to MDNR-FSS-006B.
- 8.4.2 Attach pre-cleaned, decontaminated sampler to a new or cleaned sampling line. Overhand knots approximately every two feet provide easier handling of the sampler. As an added equipment protection measure, the rope end opposite the sampler should be tied to the watercraft or other secure structure.
- 8.4.3 Open sampler jaws, insert the spring-loaded catch pin (Appendix B), and slowly lower sampler to the bottom of the water body. It is important to keep the sampler line vertical. Slowly lowering the sampler also minimizes disturbance of upper sediment layers.

- 8.4.4 Allow extra slack in the line following contact with the sediment to trip closure mechanism. From time to time, the spring loaded pin can become corroded preventing the sampler from closing. Vegetable shortening can be used to lubricate the pin without contaminating the samples, as would a petroleum lubricant.
- 8.4.5 Retrieve sampler and gently deposit contents into a decontaminated stainless steel bucket. If a composite is required, rinse the sampler between grabs with site water.
- 8.4.6 Mix and transfer as per Sections 8.1.3 and 8.1.4 with decontaminated spoon.
- 8.4.7 Preserve, handle, and document as per Section 9.0.
- 8.4.8 Decontaminate equipment as per Section 10.0.

## 9.0 HANDLING, PRESERVATION, DOCUMENTATION, AND TRANSPORTATION

- 9.1 For appropriate containers, volumes, and preservatives, refer to MDNR-FSS-001.
- 9.2 Collect GPS latitude and longitude data from each sample site.
- 9.3 Complete and attach sample labels to containers, place on ice, fill out chain-of-custody record, and enter information into field notebook according to MDNR-FSS-002, MDNR-FSS-003, and MDNR-FSS-004.
- 9.4 Handle and transport samples according to MDNR-FSS-018.

## 10.0 EQUIPMENT DECONTAMINATION

- 10.1 With scrub brush, thoroughly clean in phosphate-free detergent and hot tap water (when unavailable in the field, use deionized water)
- 10.2 Triple rinse with deionized water
- 10.3 Rinse with 5-10% HCL or HNO<sub>3</sub> to decontaminate metals
- 10.4 Immediately rinse with deionized water (acid will corrode equipment if it remains)
- 10.5 Rinse with acetone or isopropanol for organic decontamination (isopropanol should be used when collecting sediment for VOA or acetone analysis)
- 10.6 Air dry in a clean environment



10.7 Cover with clean aluminum foil

10.8 Dispose of waste decontamination fluids properly according to MDNR-FSS-206

## 11.0 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

QA/QC such as duplicate samples, trip blanks, or field blanks shall be conducted in accordance with the Fiscal Year Quality Assurance Project Plan (QAPP) for that particular project and MDNR-FSS-210.

## 12.0 REFERENCES

Landrum and Robbins, 1990, in *Sediments: Chemistry and Toxicity on In-Place Pollutants*, by R. Baudo, J. Geisy and H. Muntau.

USEPA 600/3-89/013, 1990. *Ecological Assessment of Hazardous Waste Sites: A Field and Laboratory Reference*.

American Public Health Association, 1995. *Standard Methods for the Examination of Water and Wastewater*; 19<sup>th</sup> edition.

Baudo, R., J. Geisy and H. Muntau. 1990. *Sediments: Chemistry and Toxicity of In-Place Pollutants*. Lewis. 405p.

MDNR Environmental Services Program, MDNR-FSS-001, *Required/Recommended Containers, Volumes, Preservatives, Holding Times, and Special Sampling Considerations*

MDNR Environmental Services Program, MDNR-FSS-002, *Field Sheet and Chain-of-Custody Record*

MDNR Environmental Services Program, MDNR-FSS-003, *Sample Numbering and Labeling*

MDNR Environmental Services Program, MDNR-FSS-004, *Field Documentation*

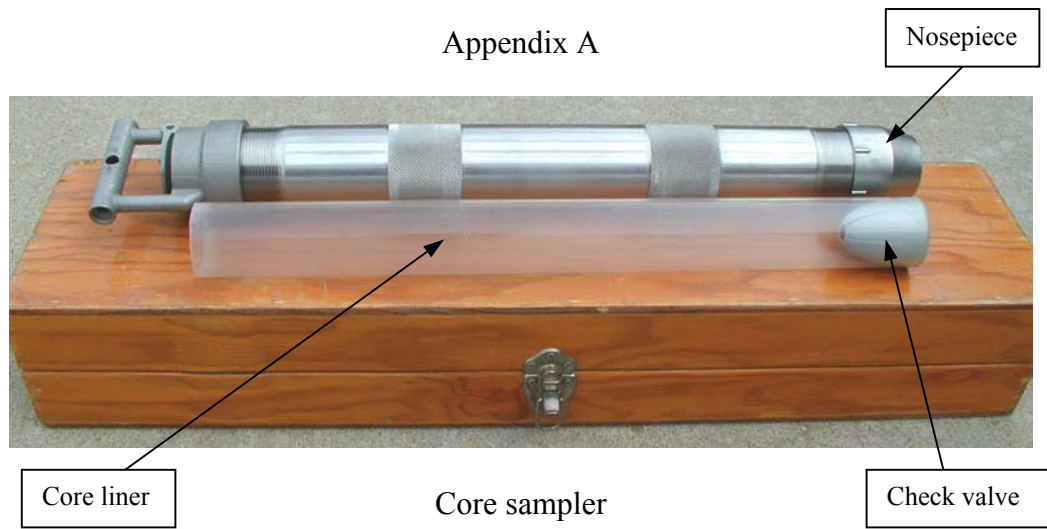
MDNR Environmental Services Program, MDNR-FSS-006B, *Sampling Soils and Other Solid Media for Volatile Organic Analysis (VOA)*

MDNR Environmental Services Program, MDNR-FSS-206, *Decontamination Procedures for Sampling Equipment in the Field or Laboratory*

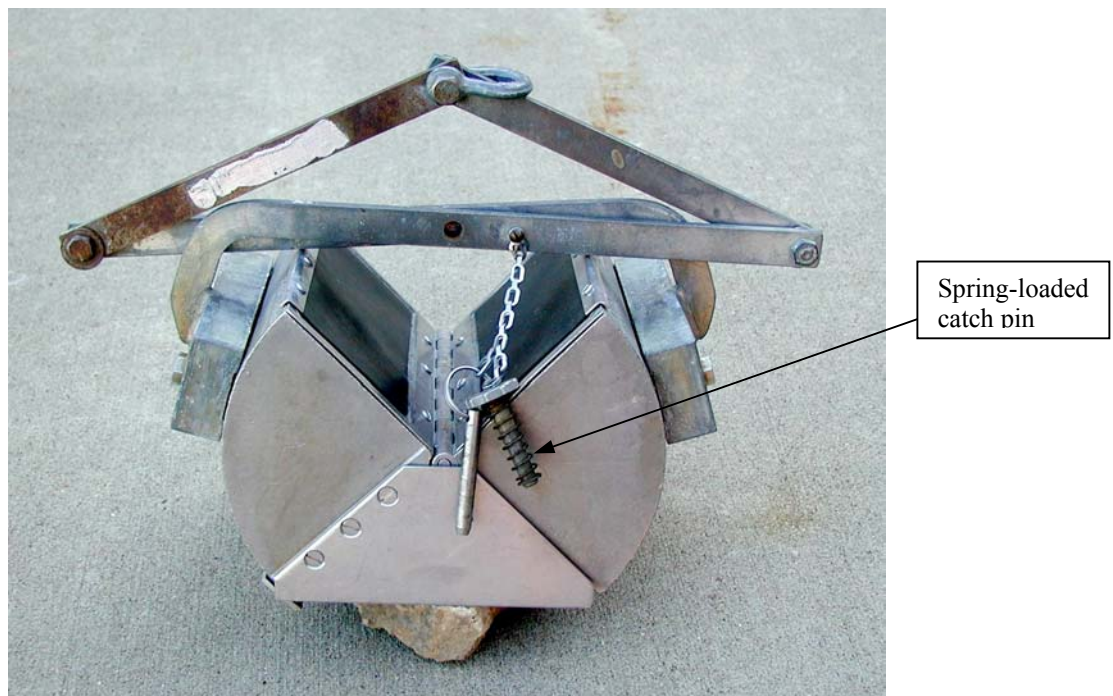
MDNR Environmental Services Program, MDNR-FSS-210, *Quality Assurance/Quality Control for Environmental Data Collection*

MDNR Environmental Services Program, MDNR-FSS-018, *Sample Handling: Field Handling, Transportation, and Delivery to the ESP Lab*

Appendix A



Appendix B



Petite Ponar